

ORIGINAL ARTICLE

Effect of dentin pre-treatment with a casein phosphopeptide-amorphous calcium phosphate (CPP-ACP) paste on dentin bond strength in tridimensional cavities

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Abstract

Objective. This study aimed to evaluate the push-out bond strength of dimethacrylate (Clearfil SE Bond/Filtek Z250; and Adper SE Plus/Filtek Z250) and silorane-based (Filtek P90 adhesive system/Filtek P90 composite resin) restorative systems following selective dentin pre-treatment with a CPP-ACP-containing paste (MI Paste). **Materials and methods.** Sixty bovine incisors were utilized. The buccal surface was wet-ground to obtain a flat dentin area. Standardized conical cavities were then prepared. Adhesive systems were applied according to manufacturers' directions and the composites were bulk-inserted into the cavity. The push-out bond strength test was performed at a universal testing machine (0.5 mm/min) until failure; failure modes were analyzed by scanning electron microscopy. Data were analyzed by two-way ANOVA and Tukey post-hoc test ($p < 0.05$). **Results.** For Clearfil SE Bond/Filtek Z250 and Filtek P90 adhesive system/Filtek P90 composite resin, the dentin pre-treatment did not influence bond strength means. For Adper SE Plus/Filtek Z250, dentin samples treated with MI Paste had statistically higher bond strength means than non-treated specimens. Adhesive failures were more frequent. **Conclusion.** Dentin pre-treatment with the CPP-ACP containing paste did not negatively affect bond strength for Clearfil SE Bond/Filtek Z250 and Filtek P90 adhesive system/Filtek P90 composite resin restorative systems and improved bond strength for the Adper SE Plus/Filtek Z250 restorative system.

Key Words: *c-Factor, low-shrinkage composite, two-step self-etching adhesive systems*

Introduction

Reliable dentin bonding systems are essential for the long-term clinical success of resin composite restorations. Adhesion is thought to enhance sealing of the cavity margin, resulting in protection of the restoration against secondary caries [1]. However, the efficacy of dentin bonding in preventing microleakage and the formation of marginal gaps is still a critical aspect of the clinical success of restorations and remains a challenge for adhesive dentistry [2]. In selecting an adhesive system for clinical use, bond

strength and sealing should play major roles. In fact, bond strength may be correlated to the ability of a restorative material to be held in place when mechanical retention is weak or missing [3].

One primary reason for the secondary caries is the lack of a tight bond between the composite resin and the underlying dentin [4]. Thus, researchers have been focused on finding an adhesive system capable of forming a strong bond to dentin and releasing protective substances, such as fluoride ions, in an attempt to reinforce the adhesive surface against a recurrent caries attack [1]. Fluoridated adhesive systems

present good dentin bonding performance and resistance to acid challenge [1,4]. Currently, other caries-preventive strategies based on the application of substances directly on dentin, such as CPP-ACP (casein phosphopeptide-amorphous calcium phosphate), have also been proposed to improve dentin resistance. CPP-ACP incorporated into a commercially available paste (MI Paste/Tooth Mousse, GC Corporation, Japan) can reduce demineralization and enhance remineralization of dentin [5–7]. Thus, the adhesive interface might present decreased susceptibility to secondary caries when pre-treated with MI Paste. However, few data have been published about the effect of dentin pre-treatment with this CPP-ACP paste on bond strength of adhesive systems, especially the two-step self-etching ones.

Traditional dimethacrylate-based two-step self-etching adhesive systems incorporate fewer application steps than the three-step total-etch adhesives, allowing simultaneous demineralization and infiltration [8] and providing satisfactory bond strength to dentin [9]. These adhesive systems are generally used with dimethacrylate-based composite resins. A recently-introduced low-shrinkage composite based on silorane monomers (Filtek P90, 3M ESPE, St. Paul, MN) uses a dedicated two-step self-etching adhesive system, the formulation of which is claimed by the manufacturer to specifically fit the chemistry of the composite. If the application of a CPP-ACP paste on dentin before two-step self-etching adhesive systems does not decrease bond strength, this adhesive protocol could be of great interest clinically.

Thus, the aim of this study was to evaluate dentin bond strength when a treatment with the CPP-ACP based MI Paste (GC Corporation, Tokyo, Japan) was performed before the placement of dimethacrylate- and silorane-based restorative systems. The hypothesis tested is that dentin pre-treatment with MI Paste does not affect bond strength of the restorative systems.

Materials and methods

Experimental design

The factors under study were (1) *dentin pre-treatment with a CPP-ACP paste* (MI Paste, GC Corporation, Tokyo, Japan) at two levels: no pre-treatment and dentin pre-treatment with MI Paste; and (2) *restorative system* at three levels: Clearfil SE Bond adhesive system (Kuraray, Tokyo, Japan) with Filtek Z250 composite resin (3M ESPE, St. Paul, MN); Adper SE Plus adhesive system (3M ESPE) with Filtek Z250 composite resin (3M ESPE); and Filtek P90 adhesive system (3M ESPE) with Filtek P90 composite resin (3M ESPE). The materials' compositions, manufacturers and batch numbers are shown in Table I.

Specimens' preparation

Sixty bovine incisors free from cracks or structural defect were selected. The teeth were disinfected in 0.1% aqueous solution of thymol at 40°C for no more than 1 week. After removing the root portions with double-face diamond saws (KG Sorensen, Barueri, SP, Brazil) (Figure 1A), the buccal aspect was wet-ground with 400 and 600 grit SiC abrasive papers in the polishing machine APL4 (Arotec, Cotia, SP, Brazil) (Figure 1B) to obtain flat surfaces in dentin. Standardized conical cavities (2 mm top diameter × 1.5 mm bottom diameter × 2 mm high) were then prepared, using #3131 diamond burs (KGSorensen, Barueri, SP, Brazil) at high-speed, under air-water cooling (Figure 1C and D). A custom-made preparation device allowed the cavity dimensions to be standardized [10]. The burs were replaced after every five preparations. In order to expose the bottom surface of the cavities, the lingual faces were ground, following the same procedure described for flattening the buccal aspects (Figure 1E). By following these procedures, a cavity with a C-factor magnitude of 2.2 was obtained [11].

The prepared specimens were assigned to six groups ($n = 10$), according to the factors under study (dentin pre-treatment × restorative system). In half of the samples ($n = 30$), 0.1 mL of MI Paste was actively applied with a brush in the cavity walls for 3 min and the excess was removed with absorbent paper and the dentin was kept moist. Each adhesive system was applied following manufacturers' directions:

- Clearfil SE Bond: One coat of the self-etching primer was applied on dentin (previously treated with MI Paste or not) with slight agitation (20 s) and air dried (10 s at 20 cm). One coat of the bonding agent was applied (15 s), air thinned (10 s at 20 cm) and light cured for 10 s.
- Adper SE Plus: One coat of the Liquid A (primer) was applied on dentin (previously treated with MI Paste or not) with slight agitation (20 s) until the red color disappeared quickly and air dried (10 s at 20 cm). One coat of Liquid B (bonding agent) was applied, air thinned and light cured for 10 s.
- Filtek P90: One coat of the self-etch primer was applied on dentin (previously treated with MI Paste or not) with slight agitation (15 s), air dried (10 s at 20 cm) and light cured for 10 s using the Coltolux LED (Coltène/Whaledent, Allstätten, Switzerland) at 1264 mW/cm². The bonding agent was applied, gently air thinned (10 s at 20 cm) and light cured for 10 s.

After the application of the adhesive systems, the specimens were placed onto a glass slab and the restorative procedures were carried out using the composites that were bulk inserted into the cavity from its wider side (Figure 1F). The photoactivation was carried out

Table I. Materials used in this study.

Material (manufacturer)	Type	Batch number	Composition by weight (%)
MI Paste (GC Corporation, Tokyo, Japan)	CPP-ACP containing paste	090406M	Glycerol (10–20), CPP-ACP (5–10), D-sorbitol (0–5), propylene glycol (0–2), silicon dioxide (0–2), titanium dioxide (0–2)
Clearfil SE Bond (Kuraray, Tokyo, Japan)	Dimethacrylate-based two-step self-etching adhesive system	Primer: 00896A Bond: 01321A	Primer: HEMA (10–30), 10-MDP (nf), hydrophilic dimethacrylate (nf), water (nf), accelerators (nf), dyes (nf), camphorquinone (nf) Bond: Bis-GMA (25–45), HEMA (20–40), 10-MDP (nf), hydrophilic dimethacrylate (nf), colloidal silica (nf), initiators (nf), accelerators (nf), dyes (nf), camphorquinone (nf)
Adper SE Plus (3M ESPE, St. Paul, MN)	Dimethacrylate-based two-step self-etching adhesive system	Primer: 00896A Bond: 01320A	Primer: water (70–80), HEMA (10–20) Bond: surface treated zirconia (15–25), TEGDMA (15–25), Di-HEMA phosphates (10–20), phosphoric acids-6-methacryloxy-hexylesters (5–10), Di-UDMA (1–10), TMPTMA (5–15), ethyl 4-dimethyl aminobenzoate (<2), camphorquinone (<2)
Filtek P90 System (3M ESPE, St. Paul, MN)	Silorane-based two-step self-etching adhesive system	Primer: N130675 Bond: N137817	Primer: HEMA (15–25), Bis-GMA (15–25), phosphoric acid-methacryloxy-hexylesters (5–15), ethanol (10–15), water (10–15), silane treated silica (8–12), 1,6-hexanediol dimethacrylate (5–10), (dimethylamino)ethyl methacrylate (<5), copolymer of acrylic and itaconic acid (<5), phosphine oxide (<5), camphorquinone (<5) Bond: substituted dimethacrylate (70–80), silane treated silica (5–10), TEGDMA (5–10), phosphoric acids-6-methacryloxy-hexylesters (<5), 1,6-hexanediol dimethacrylate (<3), camphorquinone (<3)
Filtek Z250 (3M ESPE, St. Paul, MN)	Dimethacrylate-based composite resin	N102777	Silane treated silica (75–85), Bis-EMA (1–10), UDMA (1–10), Bis-GMA (1–10), TEGDMA (<5)
Filtek P90 (3M ESPE, St. Paul, MN)	Silorane-based composite resin	N110333	Silane treated quartz (60–70), 3,4-epoxycyclohexylcyclopolymethylsiloxane (5–15), Bis-3,4-epoxycyclohexylethyl-phenylmethylsilane (5–15), yttrium trifluoride (5–15), products (<26)

CPP-ACP, Casein phosphopeptide-amorphous calcium phosphate; HEMA, 2-hydroxyethyl methacrylate; 10-MDP, 10-methacryloyloxydecyl dihydrogen phosphate; Bis-GMA, bisphenol-glycidyl methacrylate; TEGDMA, triethylene glycol dimethacrylate; UDMA, diurethane dimethacrylate; TMPTMA, trimethylolpropane trimethacrylate; Bis-EMA, bisphenol A polyethylene glycol diether dimethacrylate.

with the LED curing-light Colt lux LED (Coltène/Whaledent; 1264 mW/cm²) for 20 s. The light tip was positioned directly on the restoration, which had been previously covered with a mylar strip. The samples were stored in distilled water at 37°C for 24 h. Restorations were finished with Sof-Lex disks (3M ESPE) on the buccal and lingual aspects.

Push-out bond strength

The sample was positioned on top of a metallic device that had an aperture that allowed the smaller diameter of the restoration to be in contact with an aspheric device, connected to the load cell of a universal testing machine (EMIC, São José dos Pinhais, PR, Brazil). This spherical device applied a compressive force on the smaller diameter surface of the restoration

(Figure 1G), until rupture of the tooth–composite bond was achieved. The push-out test was carried out at a cross-head speed of 0.5 mm/min. The bond strength values were reported in MPa and derived by dividing the imposed force (in N) at the time of fracture by the bond area: $\pi(R1 + R2)\sqrt{(R1 + R2)^2 + h^2}$, where $R1$ represents the base radius, $R2$ represents the top radius and h represents the height of the cavity.

Scanning electron microscopy analysis

The fractured specimens were cut in half with a water-cooled low-speed diamond saw (Buehler, São Paulo, SP, Brazil) to obtain two specimens. Both specimens were fixed to aluminum stubs (Procind Ltda, Piracicaba, SP, Brazil) with the fractured interfaces facing upward, sputter-coated with gold (SDC

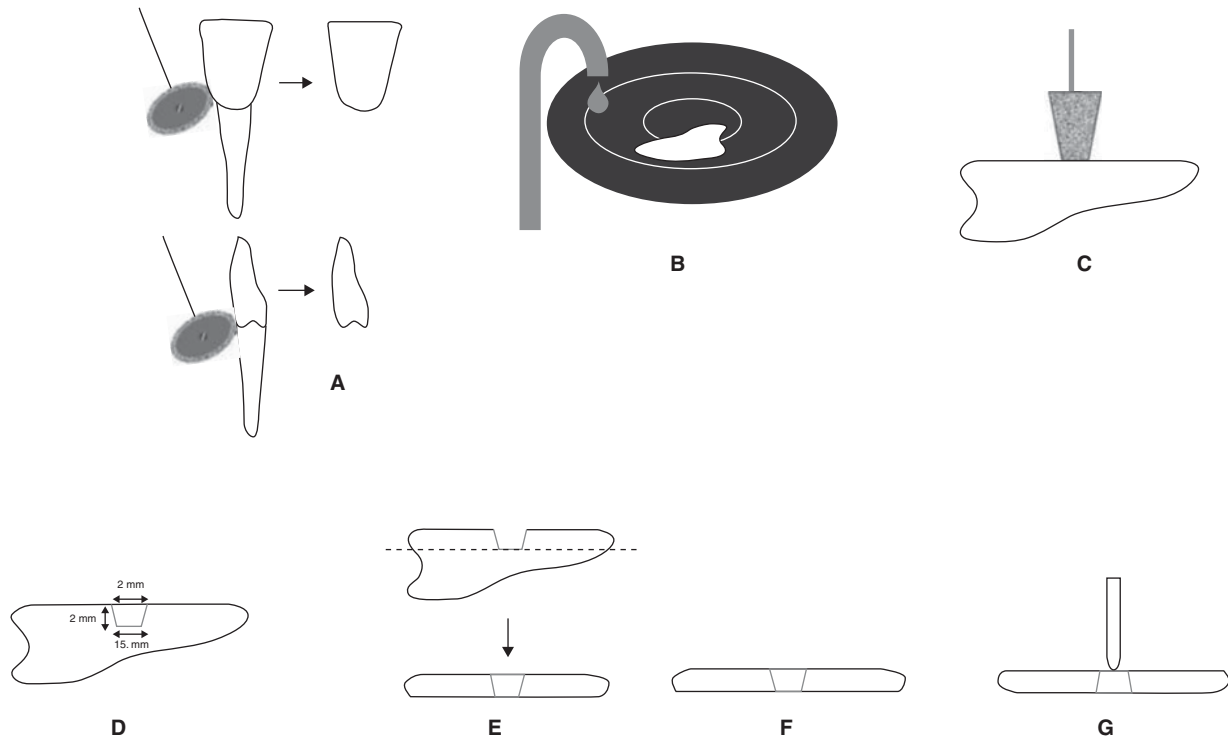


Figure 1. Schematic representation of the push-out test: (A) Frontal and lateral aspects of the roots being removed with a double-face diamond saw. (B) The vestibular surface was ground in a polishing machine to expose a flat dentin surface. (C) Cavities were prepared using #3131 diamond burs in standardized dimensions: 2 mm top diameter \times 1.5 mm bottom diameter \times 2 mm high (D). (E) The lingual surface was ground. (F) The adhesive systems were applied on dentin and cavities were bulk filled with composites. (G) Push-out test conducted in a universal test machine.

050 Suptter Coater, Baltec) and evaluated by scanning electron microscopy (JEOL, Tokyo, Japan) to determine the failure modes (Figure 2): adhesive failure, cohesive failure in composite or mixed (adhesive and cohesive in composite).

Statistical analysis

Bond strength data were subjected to two-way analysis of variance (ANOVA) and Tukey post-hoc test. Failure modes were analyzed descriptively. All statistical analysis was performed at a pre-set significance level of 5% using the ASSISTAT Software (Federal University of Campina Grande, Campina Grande, Brazil).

Results

Bond strength

There were statistically significant differences between dentin pre-treatments, adhesive systems and in the interaction dentin pre-treatment \times restorative system. Comparison among the groups is shown in Table II. For Clearfil SE Bond/Filtek Z250 and Filtek P90 adhesive system/Filtek P90 composite resin restorative systems, the dentin pre-treatment did not influence bond strength means. For Adper SE Plus/Filtek Z250, dentin samples treated with MI

Paste had statistically higher bond strength means than did the non-treated samples. The adhesive systems showed statistically-similar bond strength means when dentin had no pre-treatment. For samples pre-treated with MI Paste, Filtek P90 adhesive system/Filtek P90 composite resin showed the highest bond strength means, whereas Clearfil SE Bond/Filtek Z250 had the lowest bond strength means.

Failure modes

The failure modes are displayed in Table III. The most prevalent failure mode was the adhesive one. For Adper SE Plus, samples pre-treated with MI Paste presented a lower frequency of adhesive failures and a higher frequency of cohesive failures in composite than did the non-treated samples.

Discussion

An effective bond between tooth/composite must present adequate bond strength and compensate for the shrinking and polymerization stress of the composite resin [12]. Different methods have been used to measure composite resin bond strength, such as shear bond strength and tensile bond strength. One disadvantage of these methodologies is that the tests are generally performed on flat surfaces. In such situations, the C-factor is very low and the development

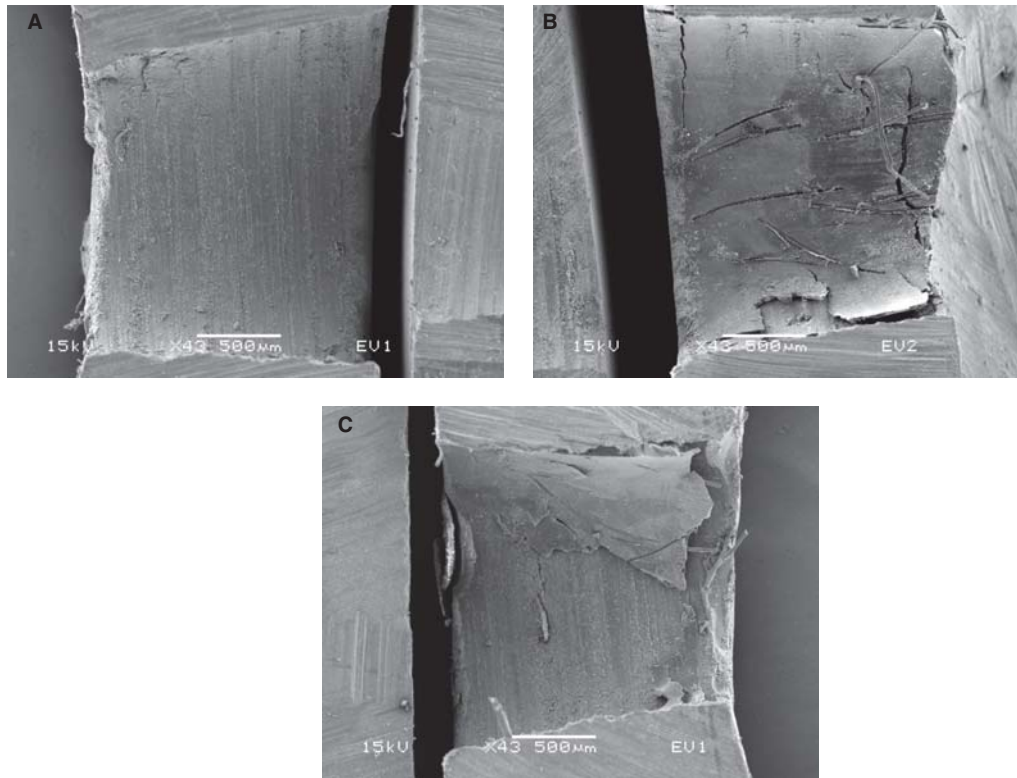


Figure 2. Failure modes obtained in this study: (A) Adhesive failure. (B) Cohesive failure in composite. (C) Mixed (adhesive and cohesive in composite).

of shrinkage stress is not directed to the bonding interface [10,13]. In this sense, the present study used the push-out bond strength test to evaluate adhesion of composite resin to coronal dentin. The advantage of using the push-out test was that the bond strength could be evaluated in a high C-factor cavity (2.2), with high stress generation directed to the bonding area [10]. The entire bonding area was submitted to the compressive force at the same time, allowing shear bond strength to be evaluated in a cavity. The method also provides a better estimation of bond strength than does the conventional shear test, as fracture occurs in parallel (not transverse) with the dentin-

bonding interface, thereby simulating clinical conditions [14,15].

Analysis of the results revealed that push-out bond strength of the restorative systems tested was not adversely affected by dentin pre-treatment with MI Paste, thus validating the hypothesis tested. Since it has been found that the application of a CPP-ACP paste can provide a caries-preventive effect, can increase remineralization and decrease the demineralization susceptibility of dentin [5–7,16], the adhesive interface might present decreased susceptibility to secondary caries when pre-treated with MI Paste. It should be taken into account that the superimposed influence of dietary acids is known to increase tooth wear rates [17]. Also, secondary caries at the restoration margins is a frequent reason for replacement of existing restorations [18]. Thus, the findings obtained in the present investigation might be of great relevance, although further *in vitro* and *in vivo* studies should be performed to clarify these suppositions.

It has also been found that the dentin pre-treatment with a CPP-ACP paste analog to MI Paste (Tooth Mousse) does not adversely affect resin–dentin interfacial morphology [19] and dentin bonding for a two-step self-etching adhesive system [20]. However, in those investigations, the CPP-ACP paste was left in place on dentin for 60 min per day and this application was repeated daily for 7 days. Considering a restorative procedure, the

Table II. Bond strength means (standard deviation) for each adhesive system according to the dentin pre-treatment.

Restorative systems	Dentin pre-treatment	
	No	MI Paste
Clearfil SE Bond/ Filtek Z250	12.48 (3.86) aA	13.54 (4.95) bA
Adper SE Plus/ Filtek Z250	9.37 (3.45) aB	16.55 (4.69) abA
Filtek P90 adhesive system/Filtek P90 composite resin	14.69 (7.5) aA	18.60 (5.22) aA

Different letters (lower case–column, upper case–row) differ by Tukey test at the 5% confidence level.

Table III. Frequency of the failure modes.

Restorative systems	Dentin pre-treatment	Failure modes		
		A	CC	M
Clearfil SE Bond/ Filtek Z250	No MI Paste	5 5	1 2	4 3
Adper SE Plus/ Filtek Z250	No MI Paste	9 4	— 4	1 2
Filtek P90 adhesive system/Filtek P90 composite resin	No MI Paste	6 6	2 2	2 2

A, adhesive failure; CC, cohesive failure in composite; M, mixed (adhesive and cohesive in composite).

adhesive system should be applied into the cavity immediately after tooth preparation. Thus, the procedure above described to treat dentin with a CPP-ACP paste is not clinically viable. On the other hand, this study highlights the possibility of using a shorter time (3 min) to actively apply MI Paste immediately after cavity preparation, which can be applicable for clinicians as a novel step for adhesive procedures.

Samples treated with MI Paste prior to the application of Adper SE Plus presented statistically-improved bond strength compared to non-treated samples, as confirmed by the lower frequency of adhesive failures and the higher frequency of cohesive failures in composite. Adper SE Plus presents phosphoric acid esters that are capable of bonding chemically to hydroxyapatite [21]. Thus, this adhesion potential could have been improved by increased calcium availability after dentin treatment with MI Paste, increasing bond strength means for Adper SE Plus. On the other hand, improved bond strength after dentin pre-treatment with MI Paste would be expected for Clearfil SE Bond that contains 10-MDP. The interaction between 10-MDP and calcium would generate a stable Ca-monomer salt [22], which is responsible for high dentin bond strength values [23]. Nevertheless, dentin pre-treatment with MI Paste did not influence adhesion of Clearfil SE Bond, presuming that a low amount of Ca-monomer salt was probably produced. Further chemical analyses are necessary to confirm this assumption.

It is important to emphasize that the P90 adhesive system also contains phosphoric acid esters as well as Adper SE Plus. The treatment of samples with MI Paste and restored with P90 composite resin did not yield statistically increased bond strength in comparison with non-treated samples, although specimens restored with a P90 restorative system have shown statistically the highest bond strength means among treated samples. This finding may be due to the low-shrinkage behavior of P90 composite resin. The application of MI Paste on dentin before priming, bonding and filling the cavity with the P90 system

associated with lower stresses generated in the adhesive interfaces might have provided the highest bond strength. Further *in vitro* and *in vivo* studies should be performed to evaluate bonding durability of the novel adhesive technique described in this study.

Therefore, dentin pre-treatment with the CPP-ACP containing paste did not negatively affect bond strength for Clearfil SE Bond/Filtek Z250 and Filtek P90 adhesive system/Filtek P90 composite resin restorative systems. Bond strength values for the Adper SE Plus/Filtek Z250 restorative system increased after applying the CPP-ACP containing paste.

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References

- [1] Iida Y, Nikaido T, Kitayama S, Takagaki T, Inoue G, Ikeda M, et al. Evaluation of dentin bonding performance and acid-base resistance of the interface of two-step self-etching adhesive systems. *Dent Mater J* 2009;28: 493–500.
- [2] De Munck J, Van Landuyt K, Peumans M, Poitevin A, Lambrechts L, Braem M, et al. A critical review of the durability of adhesion to tooth tissue: methods and results. *J Dent Res* 2005;84:118–32.
- [3] Heintze SD, Thunpithayakul C, Armstrong SR, Rousson V. Correlation between microtensile bond strength data and clinical outcome of Class V restorations. *Dent Mater* 2011; 27:114–25.
- [4] Sonbul H, Birkhed D. Risk profile and quality of dental restorations: a cross-sectional study. *Acta Odontol Scand* 2010;68:122–8.
- [5] Oshiro M, Yamaguchi K, Takamizawa T, Inage H, Watanabe T, Irokawa A, et al. Effect of CPP-ACP paste on tooth mineralization: an FE-SEM study. *J Oral Sci* 2007;49: 115–20.
- [6] Yamaguchi K, Miyazaki M, Takamizawa T, Inage H, Kurokawa H. Ultrasonic determination of the effect of casein phosphopeptide-amorphous calcium phosphate paste on the demineralization of bovine dentin. *Caries Res* 2007;41:204–7.
- [7] Rahiotis C, Vougiouklakis G. Effect of a CPP-ACP agent on the demineralization and remineralization of dentine *in vitro*. *J Dent* 2007;35:695–8.
- [8] Tay FR, King NM, Chan KM, Pashley DH. How can nanoleakage occur in self-etching adhesive systems that demineralize and infiltrate simultaneously? *J Adhes Dent* 2002;4:255–69.
- [9] Erhardt MC, Pisani-Proença J, Osorio E, Aguilera FS, Toledano M, Osorio R. Influence of laboratory degradation methods and bonding application parameters on microTBS of self-etch adhesives to dentin. *Am J Dent* 2011;24:103–8.
- [10] Cunha LG, Alonso RC, Pfeifer CS, de Góes MF, Ferracane JL, Sinhorette MA. Effect of irradiance and light source on contraction stress, degree of conversion and push-

- out bond strength of composite restoratives. *Am J Dent* 2009; 22:165–70.
- [11] Cunha LG, Alonso RC, Correr GM, Brandt WC, Correr-Sobrinho L, Sinhoreti MA. Effect of different photoactivation methods on the bond strength of composite resin restorations by push-out test. *Quintessence Int* 2008;39:243–9.
- [12] Korkmaz Y, Gurgan S, Firat E, Nathanson D. Effect of adhesives and thermocycling on the shear bond strength of a nano-composite to coronal and root dentin. *Oper Dent* 2010;35:522–9.
- [13] Alonso RC, Cunha LG, Correr GM, Cunha Brandt W, Correr-Sobrinho L, Sinhoreti MA. Relationship between bond strength and marginal and internal adaptation of composite restorations photocured by different methods. *Acta Odontol Scand* 2006;64:306–13.
- [14] Kienanen P, Alander P, Lassila LV, Vallittu PK. Bonding of ceramic insert to a laboratory particle filler composite. *Acta Odontol Scand* 2005;63:272–7.
- [15] Cekic I, Ergun G, Uctasli S, Lassila LV. *In vitro* evaluation of push-out bond strength of direct ceramic inlays to tooth surface with fiber-reinforced composite at the interface. *J Prosthet Dent* 2007;97:271–8.
- [16] Yengopal V, Mickenautsch S. Caries preventive effect of casein phosphopeptide-amorphous calcium phosphate (CPP-ACP): a meta-analysis. *Acta Odontol Scand* 2009; 21:1–12.
- [17] Addy M, Shellis RP. Interaction between attrition, abrasion and erosion in tooth wear. *Monogr Oral Sci* 2006;20:17–31.
- [18] Xu HH, Moreau JL, Sun L, Chow LC. Nanocomposite containing amorphous calcium phosphate nanoparticles for caries inhibition. *Dent Mater* 2011;27:762–9.
- [19] Adebayo OA, Burrow MF, Tyas MJ. Resin-dentine interfacial morphology following CPP-ACP treatment. *J Dent* 2010;38: 96–105.
- [20] Adebayo OA, Burrow MF, Tyas MJ. Dentine bonding after CPP-ACP paste treatment with and without conditioning. *J Dent* 2008;36:1013–24.
- [21] Fu B, Sun X, Qian W, Shen Y, Chen R, Hannig M. Evidence of chemical bonding to hydroxyapatite by phosphoric acid esters. *Biomaterials* 2005;26:5104–10.
- [22] Yoshida Y, Nagakane K, Fukuda R, Nakayama Y, Okazaki M, Shintani H, et al. Comparative study on adhesive performance of functional monomers. *J Dent Res* 2004;83: 454–8.
- [23] Van Landuyt KL, Yoshida Y, Hirata I, Snauwaert J, De Munck J, Okazaki M, et al. Influence of the chemical structure of functional monomers on their adhesive performance. *J Dent Res* 2008;87:757–61.